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pre-exponential factor

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The thermal decomposition of several composite ablative materials being considered for shipboard use was studied using thermogravimetry. Thermograms were obtained at six heating rates ranging from 10 to 160° C/min. From these data, the kinetic parameters were determined by a modified version of Friedman's method. Activation energies determined by this method were compared with those obtained by the method of Flynn and Wall.

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FOREWORD

This work was performed as part of an investigation to screen and classify candidate ablative materials for use in shipboard applications. Funds were provided by Naval Sea Systems Command (SEA-62R) and by NSWC independent research program Task Area Number ZR000-01-01.

This work was reviewed and approved by J. J. Yagla, Head, Ship Safety Engineering Branch, and J. F. Horton, Head, Systems Safety Division.

Released by:

THOMAS A. CLARE, Head
Combat Systems Department

CONTENTS

INTRODUCTION

The rate of decomposition of an ablative material when exposed to a heat flux may be modeled by the kinetic rate equation. If it is assumed that the material dimensions remain constant, the rate equation predicts the density of the remaining char as a function of time. The ablative characteristics of the material are dependent upon both the rate of decomposition and char density. In order to predict the thermal response of the material, accurate values of the kinetic parameters over the entire range of decomposition are required for use in the rate equation. In the case of an ablative material exposed to a solid rocket motor exhaust, the heat flux may vary widely depending upon the geometry and/or type of motor. Therefore, the effect of the heating rate on the kinetic parameters must be known as well.

We report here the kinetic parameters for the thermal decomposition of several ablative materials being considered for shipboard application. These kinetic parameters were determined by a multiple heating rate thermogravimetric technique developed by Friedman[1] and later modified by Henderson, Wiebelt, Tant, and Moore[2] to improve accuracy of the kinetic model over the entire range of decomposition. This modified version of Friedman's method involves dividing the decomposition into two separate regions. While an average activation energy is determined for the entire decomposition range, separate pre-exponential factors and apparent orders of reaction are determined for the two regions of decomposition. For comparison with the value obtained by Friedman's method,[1] the activation energy was also determined by the method of Flynn and Wall.[3]

THEORY

The kinetic rate equation simply defines the rate of decomposition as the product of a temperature-dependent term and a weight fraction-dependent term, i.e.

$$\text{rate} = f_1\,(\text{temperature}) \bullet f_2\,(\text{weight fraction}) \qquad (1)$$

The temperature-dependent term is generally expressed by Arrhenius' law

$$f_1 = A \exp(-E/RT) \qquad (2)$$

where A = pre-exponential factor (min^{-1})

E = activation energy (cal/gmol)

R = gas constant (1.987 cal/gmol-K)

T = absolute temperature (K)

The form of the weight fraction-dependent term is, in general, chosen to provide a best fit of experimental data.

FRIEDMAN'S METHOD

Friedman's method was chosen for this application because the kinetic parameters are determined from data obtained over a wide range of heating rates. Clearly, parameters obtained over a range of heating rates may be applied with greater confidence at the heating rates to which ablative materials are subjected. Another advantage of this method is that the weight fraction-dependent term is an arbitrary function whose form is determined directly by experiment.

The rate equation proposed by Friedman is

$$-1/W_0 \times dW/dt = Af\,(W/W_0)\exp(-E/RT) \tag{3}$$

where W = instantaneous weight of material (mg)

W_0 = original weight of material (mg)

dW/dt = rate of weight loss (mg/min)

$f(W/W_0)$ = undefined function of weight

Taking the natural logarithm of both sides of Equation (3) results in

$$\ln[-1/W_0 \times dW/dt] = \ln[Af(W/W_0)] - E/RT \tag{4}$$

A linear equation may be fit to $\ln[-1/W_0 \times dW/dt]$ as a function of $1/T$ at constant parametric values of W/W_0. These equations will have slopes of $-E/R$, and each intercept is the value of $\ln[Af(W/W_0)]$ at the parametric value of W/W_0. The weight fraction-dependent term, $f(W/W_0)$, is then defined as

$$f(W/W_0) = [(W-W_f)/W_0]^n \tag{5}$$

where n = order of reaction

W_f = final weight of charred material (mg)

Finally, multiplying Equation (5) by A and taking the natural logarithm results in

$$\ln[Af(W/W_0)] = \ln A + n\ln[(W-W_f)/W_0] \tag{6}$$

The final weight fraction, W_f/W_0, is determined from the original thermograms. Since $\ln[Af(W/W_0)]$ is known for various W/W_0 ratios, Equation (6) can be used to determine values of A and n. As mentioned previously, Henderson et al.[2] modified this method to improve the correlation of the kinetic model and experimental data. The weight loss curve was separated into two regions, and separate pre-exponential factors and apparent orders of reaction were determined for each region. A single average activation energy was determined for the entire weight loss curve.

FLYNN AND WALL'S METHOD

The method of Flynn and Wall[3] may be used to determine the activation energy utilizing thermograms obtained at several heating rates. The activation energy is given by

$$E \approx - (R/C)\, d(\log \beta)/d(1/T) \qquad (7)$$

where β = heating rate (K/min)

$C = C(E/RT)$

If $1/T$ versus $\log \beta$ is plotted at several weight loss fractions, a series of straight lines with slope $\Delta(\log \beta)/\Delta(1/T)$ results. The activation energy can then be calculated by Equation (7) using the slope and the appropriate value of C. Since C is a function of E/RT, the calculation of E is an iterative process. Values of C over the range $7 \leqslant E/RT \leqslant 60$ are available from a table constructed by Flynn and Wall.[3] The variation of C over this range is approximately ±3 percent. The simplicity of this method makes its use for the present application quite attractive.

EXPERIMENTAL

MATERIALS

The four ablative materials studied were supplied by Fiberite Corp. and Fiber Materials, Inc. As shown in Table 1, these materials consisted of either a phenol-formaldehyde or acrylonitrile-butadiene resin with specified amounts of glass or fiberglass added as filler.

The materials were converted to powder form by machining and were then filtered through a No. 20 sieve. They were stored overnight in a vacuum dessicator maintained at 35°C to remove traces of water.

Table 1. Composition of Materials Tested

Contents	Material Composition (percent) Fiberite Corp. MXBE-350	Fiberite Corp. MXB-360	Fiber Materials, Inc. FR-1	Fiber Materials, Inc. FR-2
Glass powder (SiO_2)	15.5	14.5	–	–
Glass fiber (SiO_2)	–	–	42.0	–
Fiberglass	41.0	59.0	–	–
Carbon	–	–	–	40.0
Total filler content	56.5	73.5	42.0	40.0
Pheno-formaldehyde resin	–	26.5	58.0	60.0
Acrylonitrile-butadiene resin	43.5	–	–	–

APPARATUS AND PROCEDURE

A Perkin-Elmer TGS-2 Thermogravimetric System was used, with temperature control provided by a Perkin-Elmer System 4 Microprocessor Controller. The sample temperature was measured with a chromel-alumel thermocouple, which was calibrated with a set of five Curie standards in the temperature range of interest at each heating rate used.[4]

In order to reduce temperature gradients in the material and to ensure uniform heating, small weights of a powdered form of the materials were used. Samples weighing 7.5 ± 0.5 mg were heated from 40 to 950°C using heating rates of 10, 20, 40, 80, 100, and 160°C/min. Both the percentage of initial weight and the rate of weight loss were plotted directly as a function of temperature. The samples were maintained in a nitrogen atmosphere throughout the experiment. When the programmed temperature scan reached 950°C, the purge gas was automatically switched to oxygen to thermo-oxidatively degrade the remaining resin. To verify the initial weight fraction of filler, the temperature was held at 950°C until the resin had completely degraded.

RESULTS

In the original thermograms, both the fraction of weight remaining and the derivative of weight loss were plotted as a function of temperature. These data were digitized at 0.01 intervals of the fraction of weight remaining and the experimental temperatures were corrected using the Curie standard temperature calibration. The thermograms were then reproduced from these data. Figure 1 shows the fraction of weight remaining as a function of temperature for each material at each heating rate. Similarly, Figure 2 shows the rate of weight loss as a function of temperature for each material at each heating rate. The digitized data for all four materials are given in the Appendix.

Plots of $\ln[-1/W_0 \times dW/dt]$ versus $1/T$ are shown in Figure 3 for each of the four materials. The slope of each line was determined from a least squares fit of the data, and the corresponding activation energy and intercept $\ln[Af(W/W_0)]$ at each value of weight loss were then determined. These data are shown in Figure 4 for each material. Plots of $\ln[Af(W/W_0)]$ versus $\ln[(W-W_f)/W_0]$ are shown in Figure 5 for each material. Figure 5 clearly illustrates the separation of the decomposition into two regions and the corresponding least squares fit for each region. A separate pre-exponential factor and reaction order was determined for each of these two regions utilizing Equation (6). The average activation energies determined from Figure 4 were used for both regions. Using Flynn and Wall's method and the plots of log β versus $1/T$ shown in Figure 6, average activation energies were determined for each of the four materials. A summary of the calculated kinetic parameters for the four materials is given in Table 2.

The kinetic parameters for the thermal decomposition of the four ablative materials were used in Equation (3) to calculate the fraction of weight remaining as a function of temperature at each heating rate. Each set of parameters was applied to that portion of the weight loss curve from which it was determined. The results of these calculations at 10 and 160°C/min are shown along with experimental data in Figure 7 for comparison. The average error, standard deviation of errors, and the 95-percent confidence interval were calculated for the experimental versus calculated points for each material, and these results are presented in Table 3.

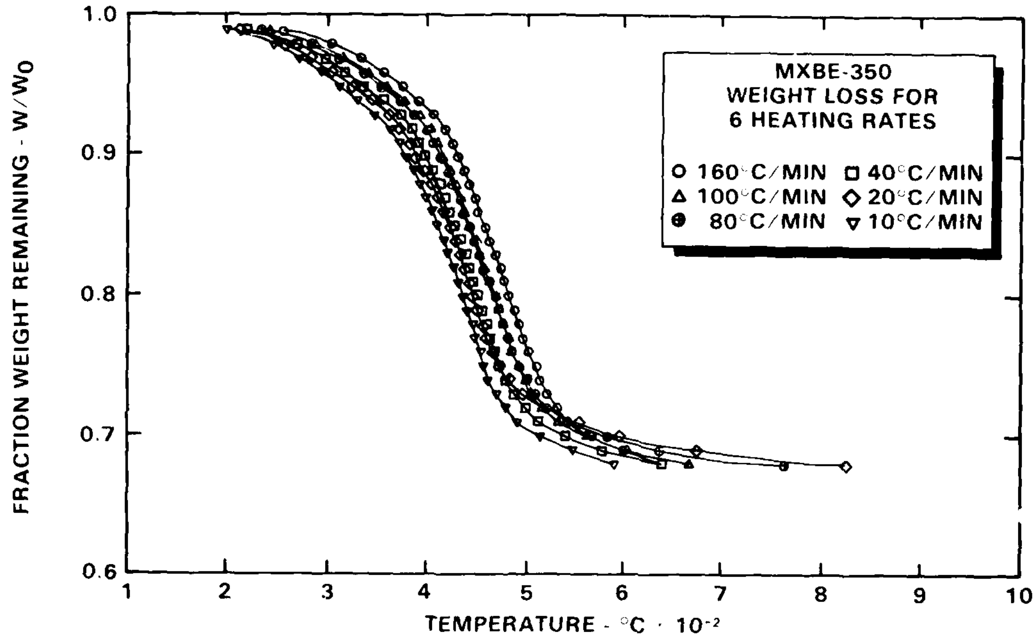

Figure 1.a Weight Loss Thermograms for MXBE-350 at Several Heating Rates

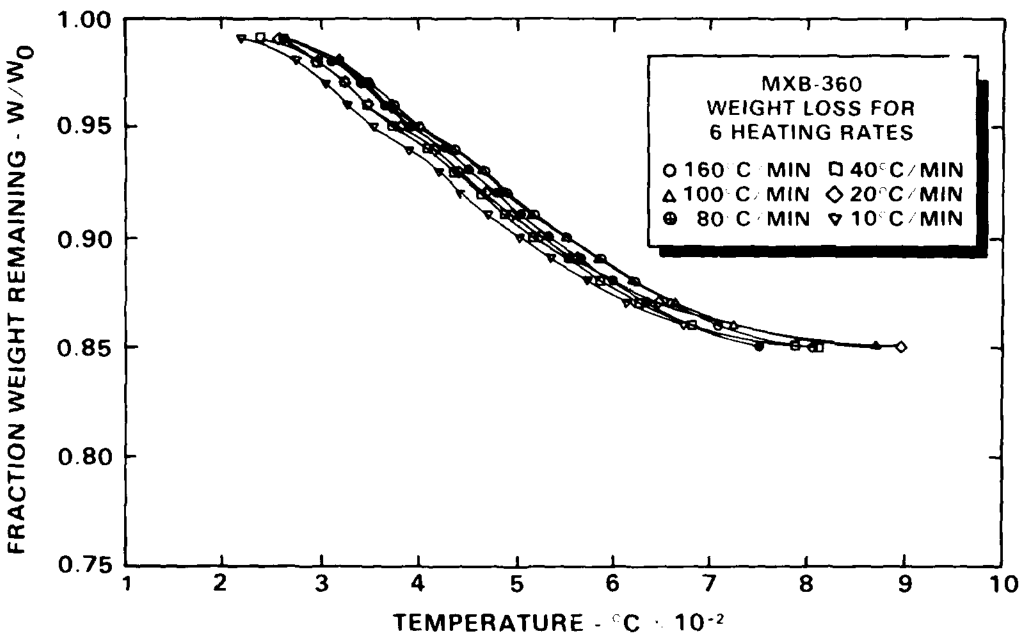

Figure 1.b Weight Loss Thermograms for MXB-360 at Several Heating Rates

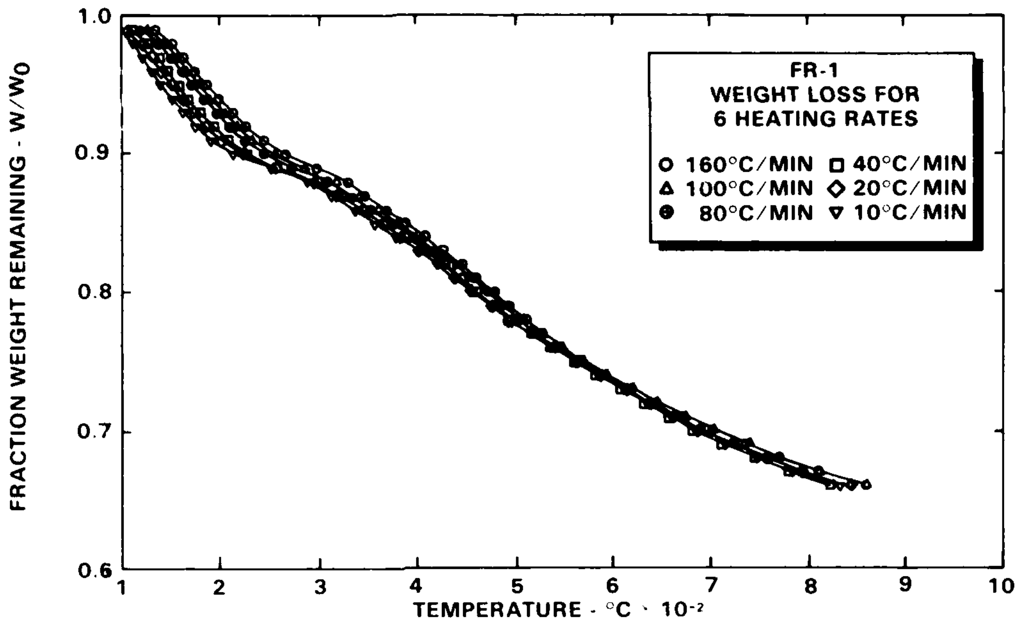

Figure 1.c Weight Loss Thermograms for FR-1 at Several Heating Rates

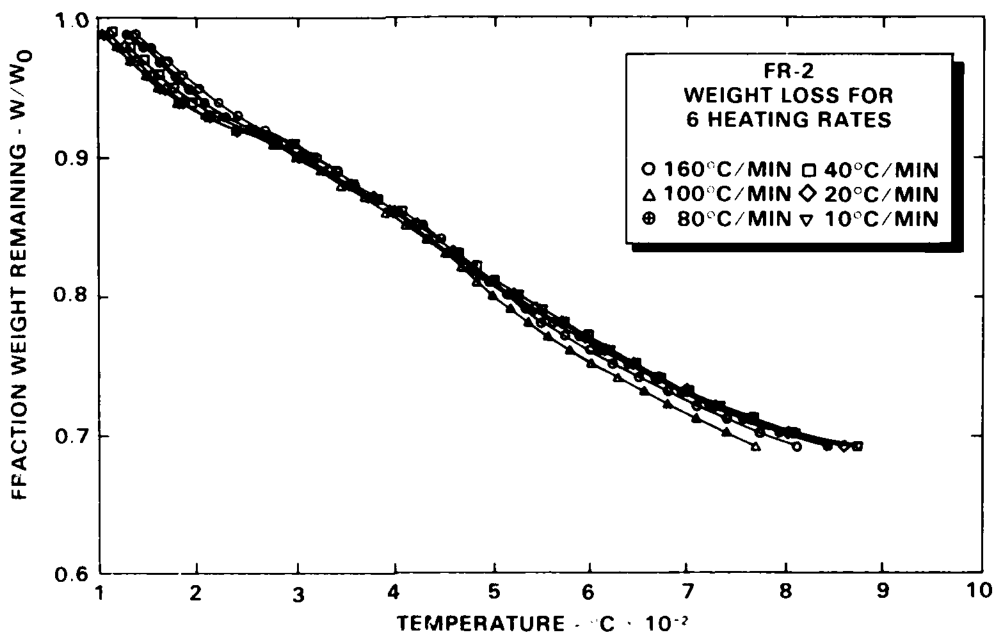

Figure 1.d Weight Loss Thermograms for FR-2 at Several Heating Rates

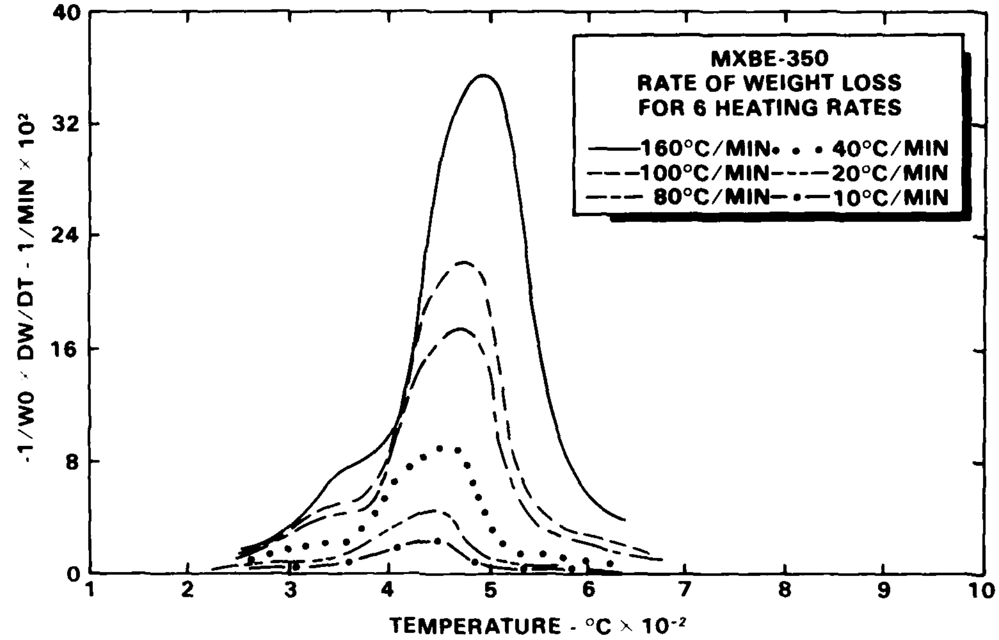

Figure 2.a Rate of Weight Loss for MXBE-350 at Several Heating Rates

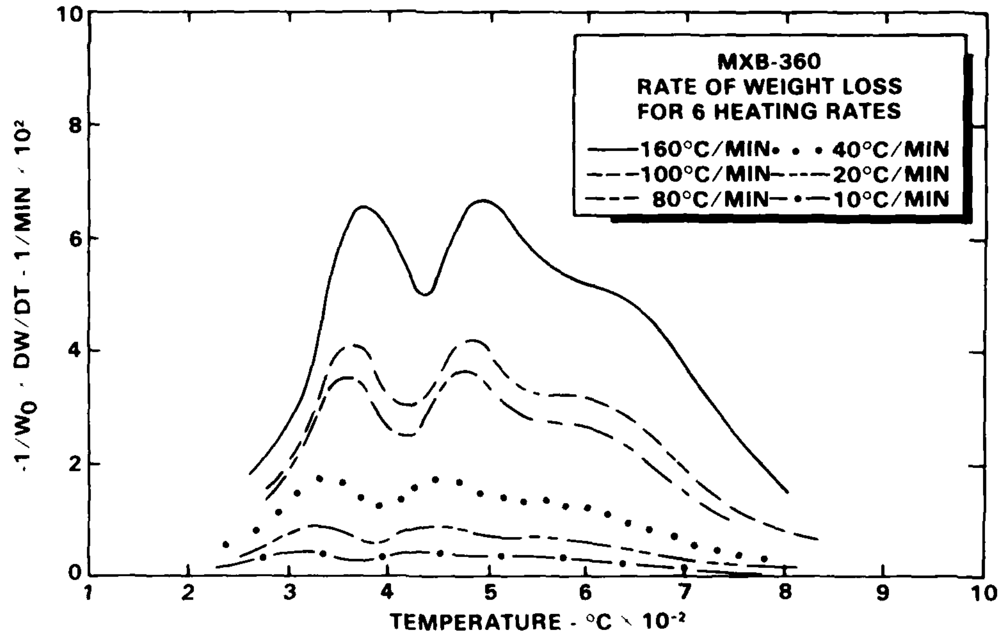

Figure 2.b Rate of Weight Loss for MXB-360 at Several Heating Rates

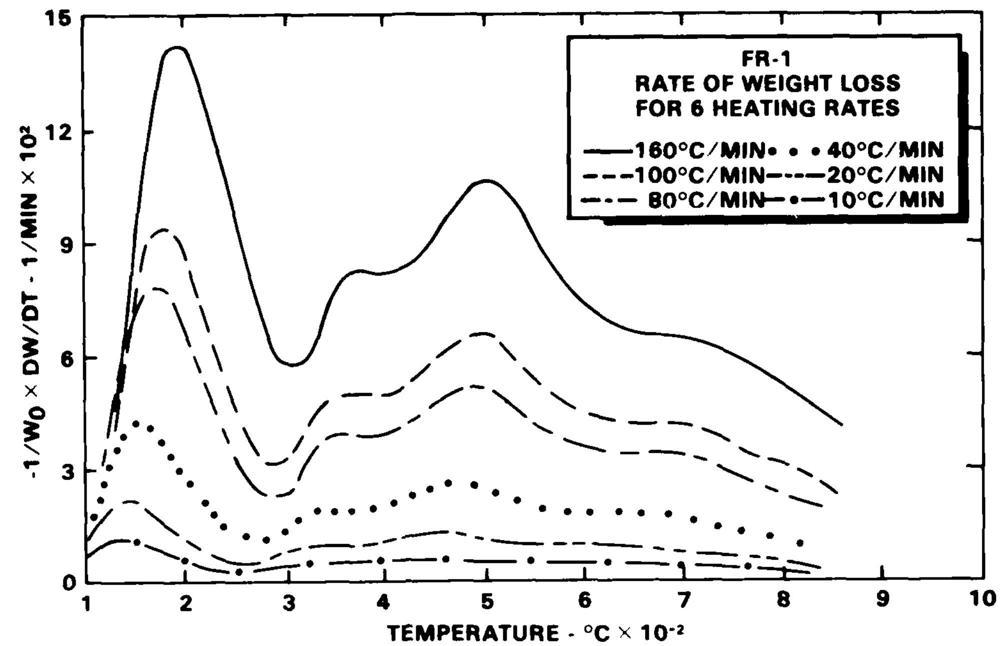

Figure 2.c Rate of Weight Loss for FR-1 at Several Heating Rates

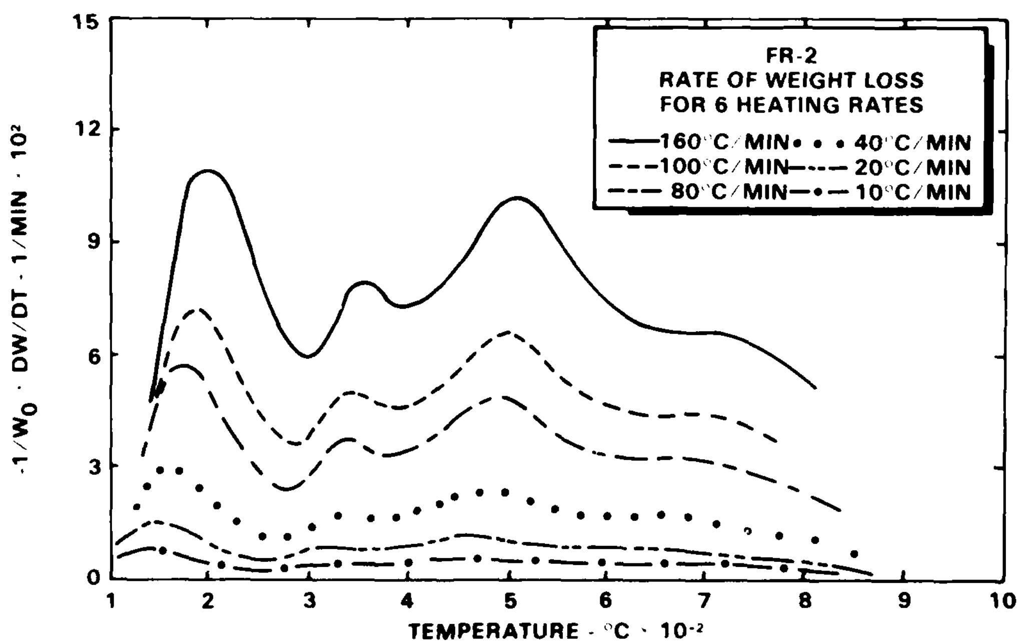

Figure 2.d Rate of Weight Loss for FR-2 at Several Heating Rates

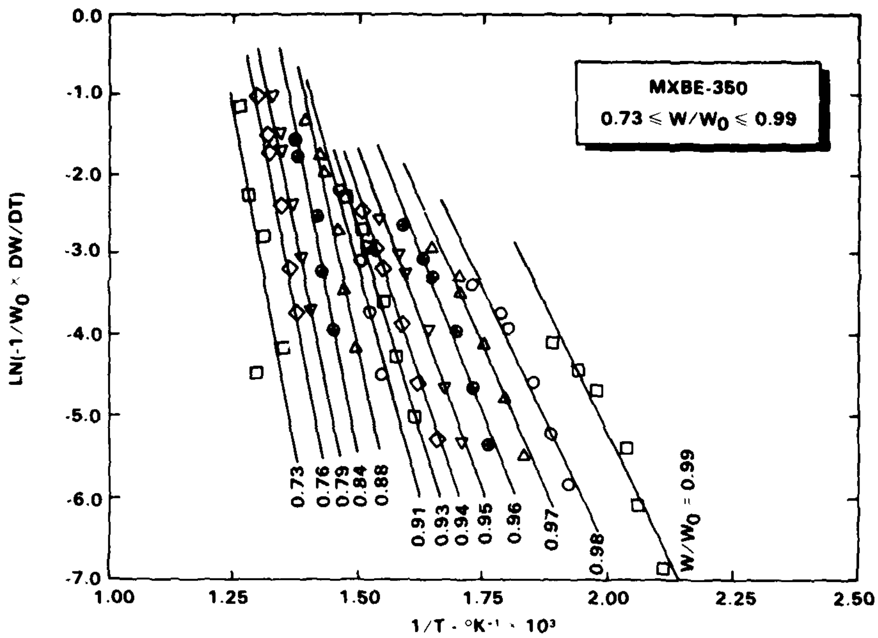

Figure 3.a Plot of Slopes Used to Determine the Activation Energy for MXBE-350

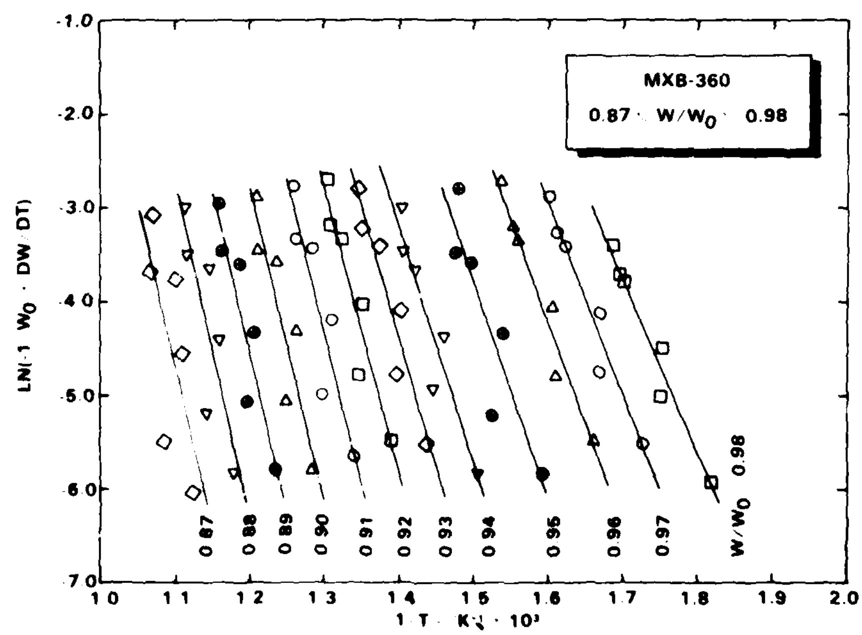

Figure 3.b Plot of Slopes Used to Determine the Activation Energy for MXB-360

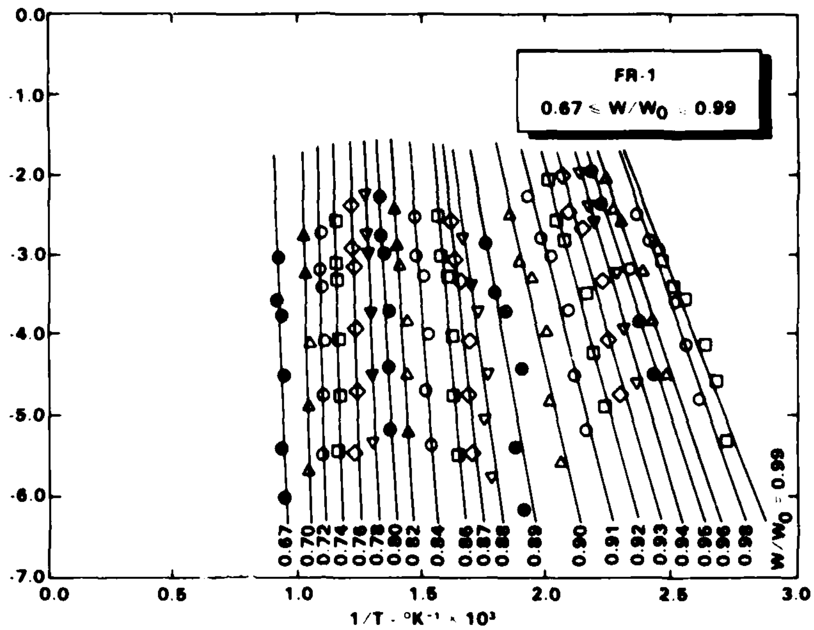

Figure 3.c Plot of Slopes Used to Determine the Activation Energy for FR-1

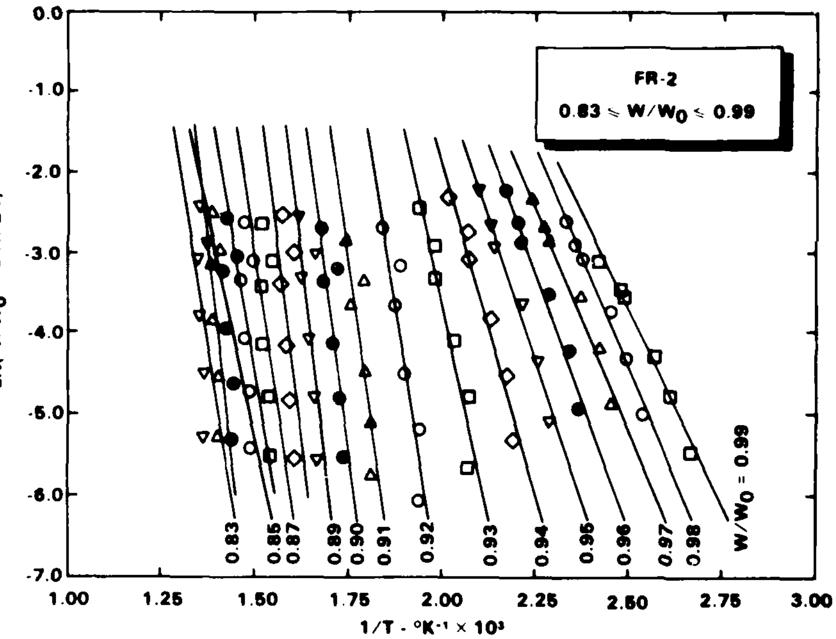

Figure 3.d Plot of Slopes Used to Determine the Activation Energy for FR-2

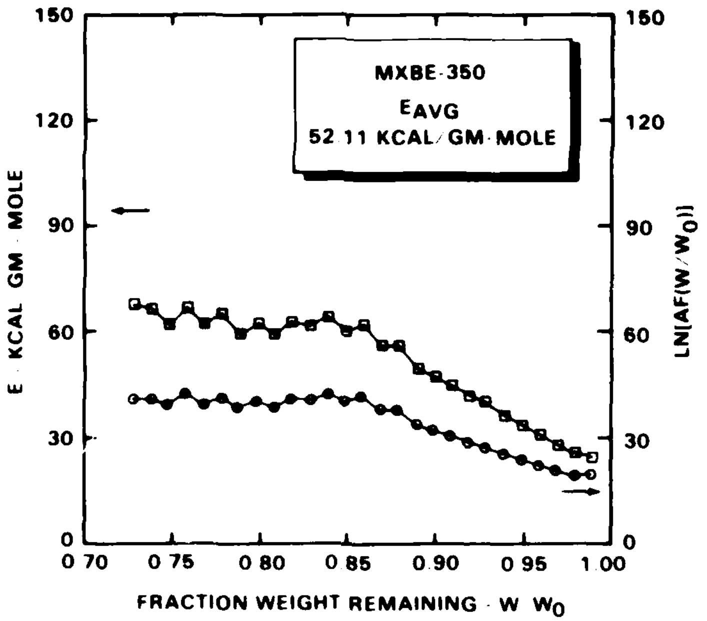

Figure 4.a Activation Energy and Intercept as a Function of Degree of Conversion for MXBE-350

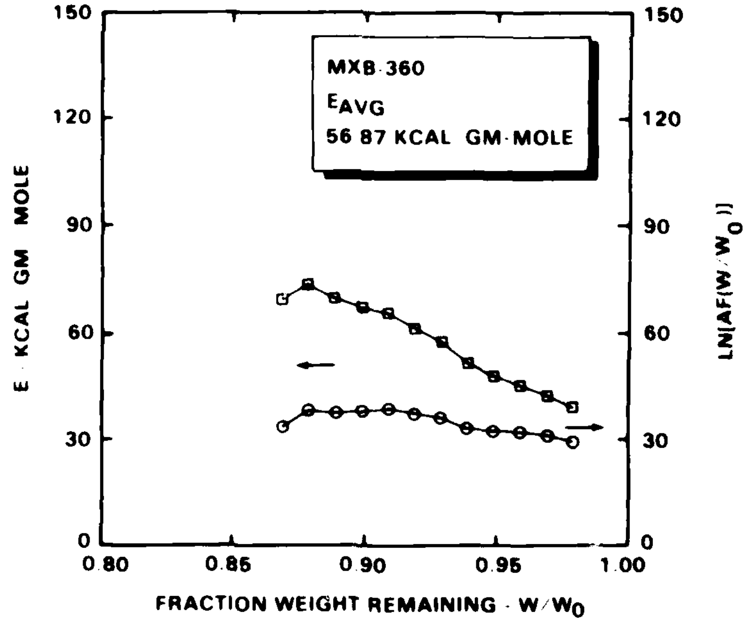

Figure 4.b Activation Energy and Intercept as a Function of Degree of Conversion for MXB-360

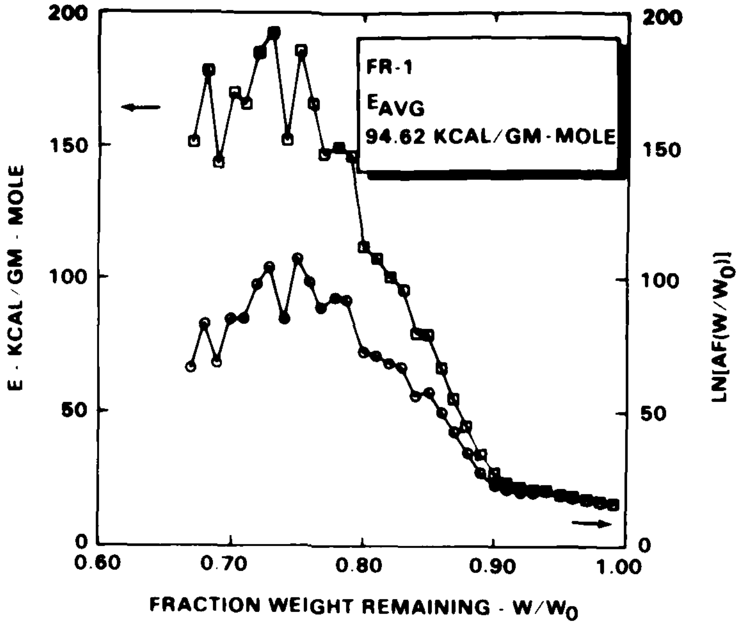

Figure 4.c Activation Energy and Intercept as a Function of Degree of Conversion for FR-1

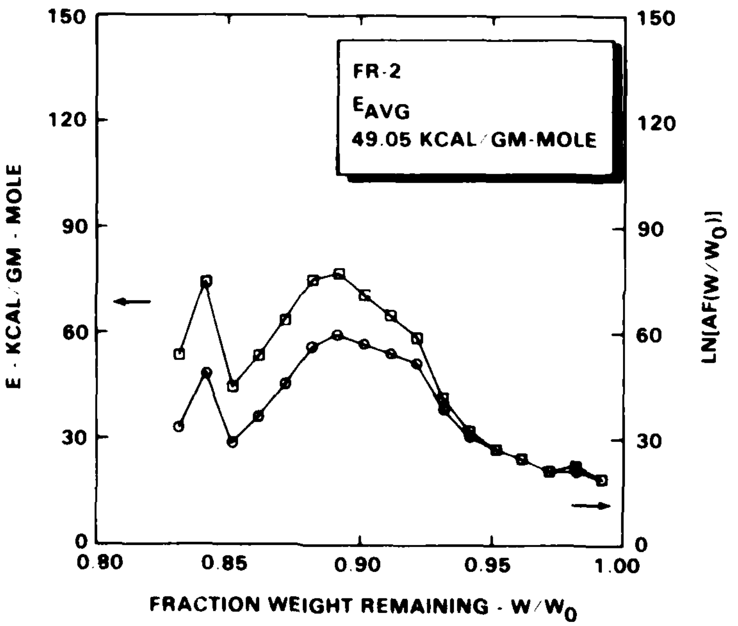

Figure 4.d Activation Energy and Intercept as a Function of Degree of Conversion for FR-2

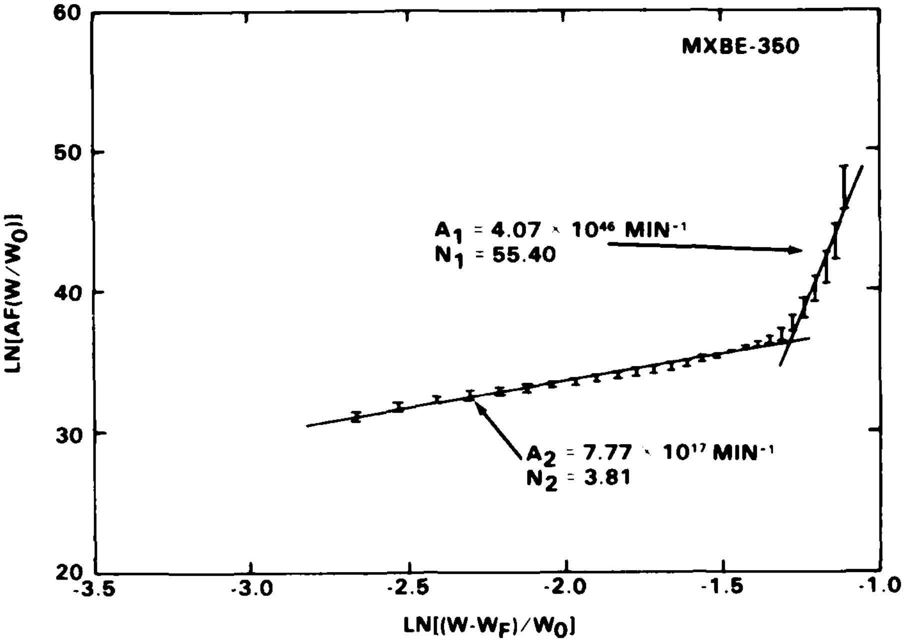

Figure 5.a Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for MXBE-350

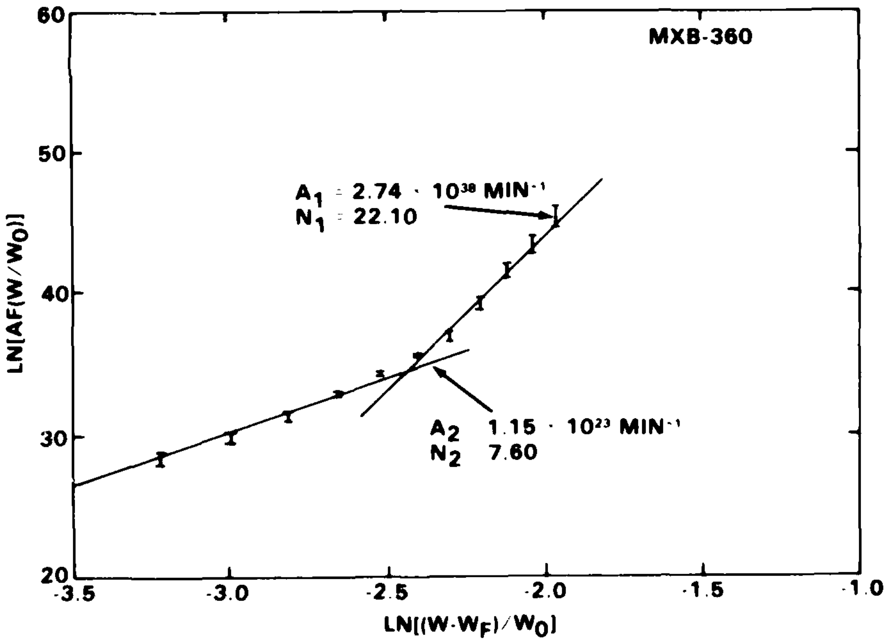

Figure 5.b Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for MXB-360

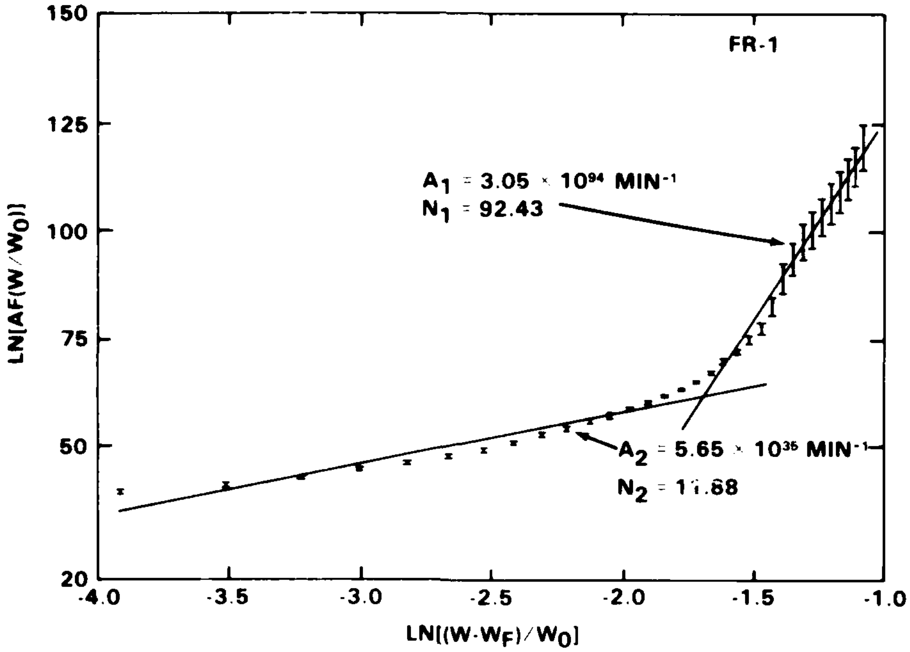

Figure 5.c Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for FR-1

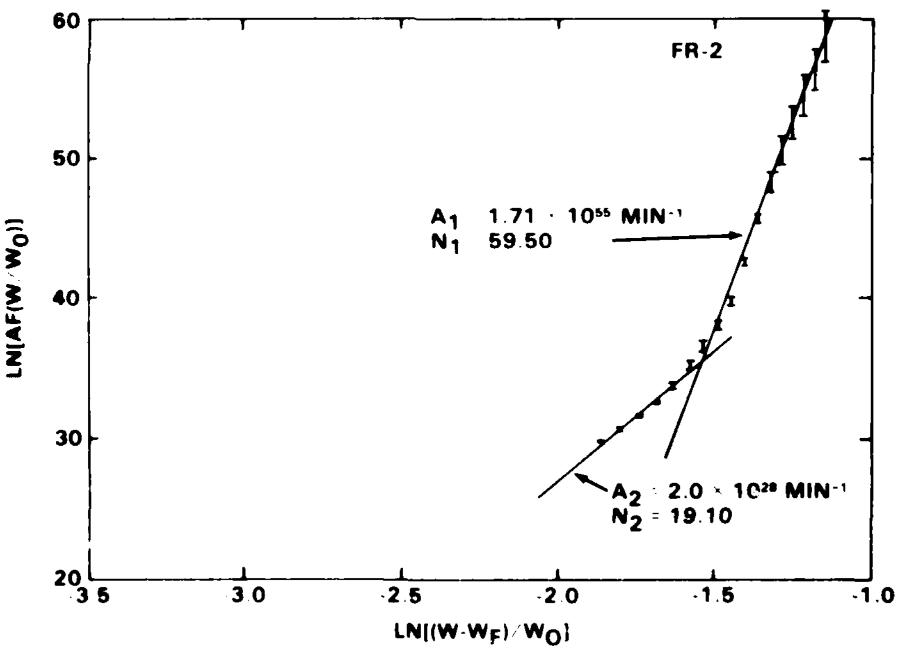

Figure 5.d Plot to Determine Pre-exponential Factor and Order of Reaction for Two Regions of Weight Loss for FR-2

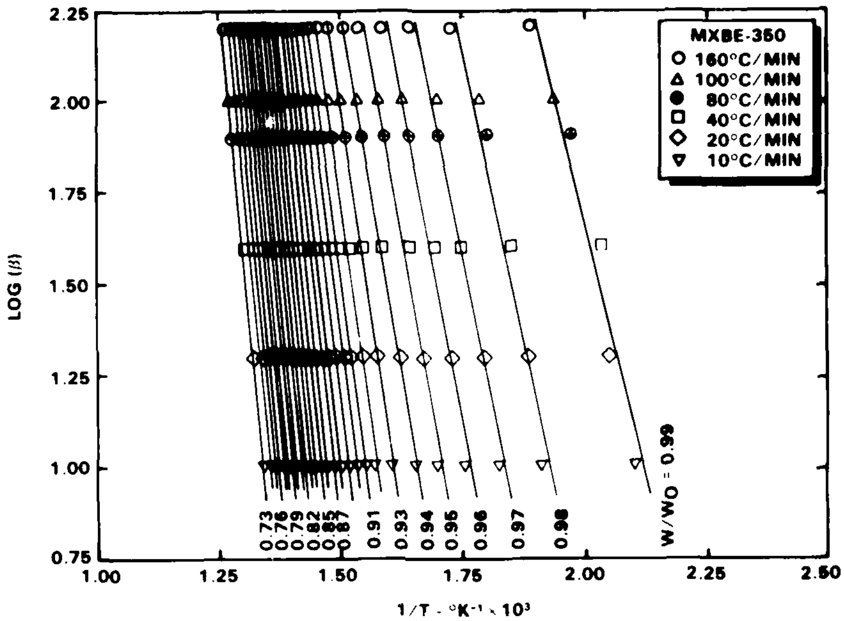

Figure 6.a Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for MXBE-350

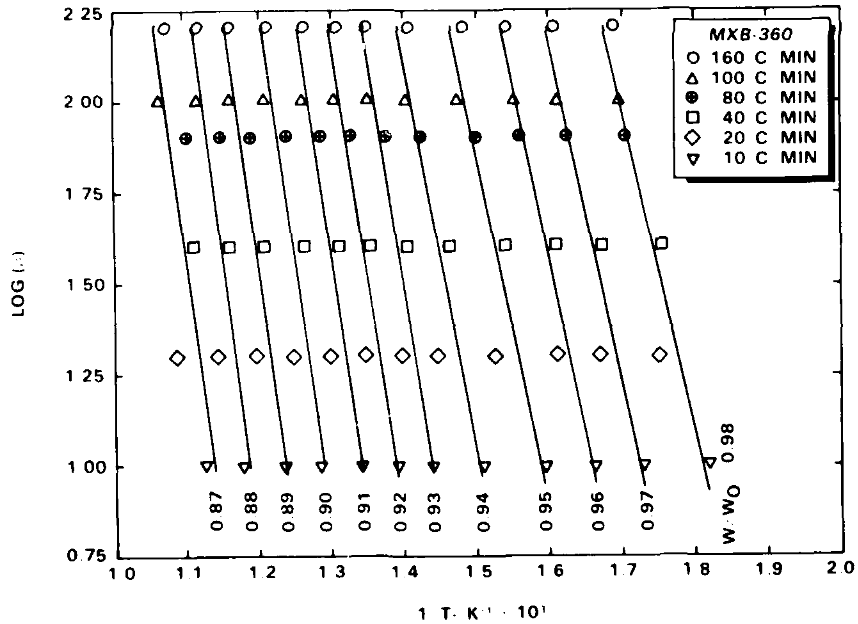

Figure 6.b Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for MXB-360

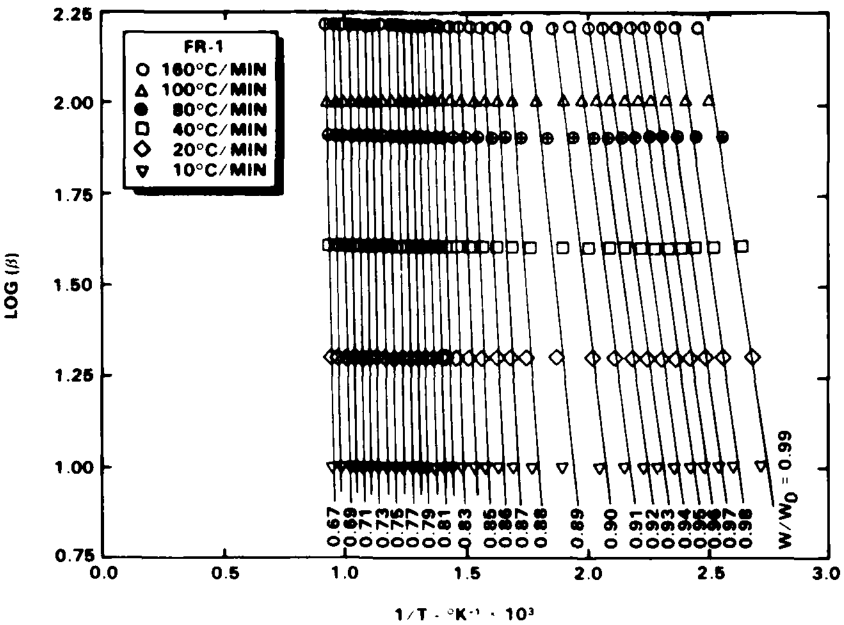

Figure 6.c Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for FR-1

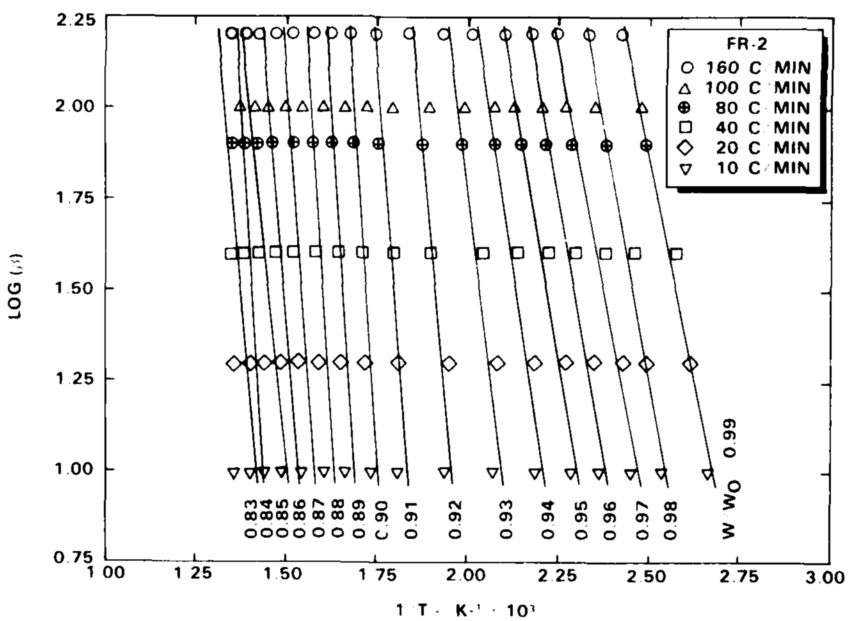

Figure 6.d Plot of Slopes Used to Determine Activation Energy by Flynn and Wall's Method for FR-2

Table 2. Results of Thermogravimetric Analysis

Material	W_f/W_0	Range of W/W_0	E_{avg} (kcal/gm-mole) Friedman	Flynn & Wall	A(min^{-1})	n	W/W_0
Fiberite MXBE-350	0.660	0.99 - 0.73	52.11	50.22	4.07×10^{46}	55.40	≥ 0.94
					7.77×10^{17}	3.81	< 0.94
Fiberite MXB-360	0.840	0.98 - 0.87	56.87	53.70	2.74×10^{38}	22.10	≥ 0.93
					1.15×10^{23}	7.60	< 0.93
Fiber materials FR-1	0.650	0.99 - 0.67	94.62	88.49	3.05×10^{94}	92.43	≥ 0.84
					5.65×10^{35}	11.88	< 0.84
Fiber materials FR-2	0.675	0.99 - 0.83	49.05	45.38	1.71×10^{55}	59.50	≥ 0.89
					2.00×10^{28}	19.10	< 0.89

Table 3. Statistical Analysis of Errors in Computed Versus Experimental W/W_0

Material	Average Error (percent)	Standard Deviation (percent)	95-Percent Confidence Interval	Number of Data Points
MXBE-350	1.36	1.41	1.17 - 1.55	162
MXB-360	0.18	0.48	0.08 - 0.29	72
FR-1	0.50	1.22	0.38 - 0.72	198
FR-2	1.07	2.30	0.74 - 1.41	90

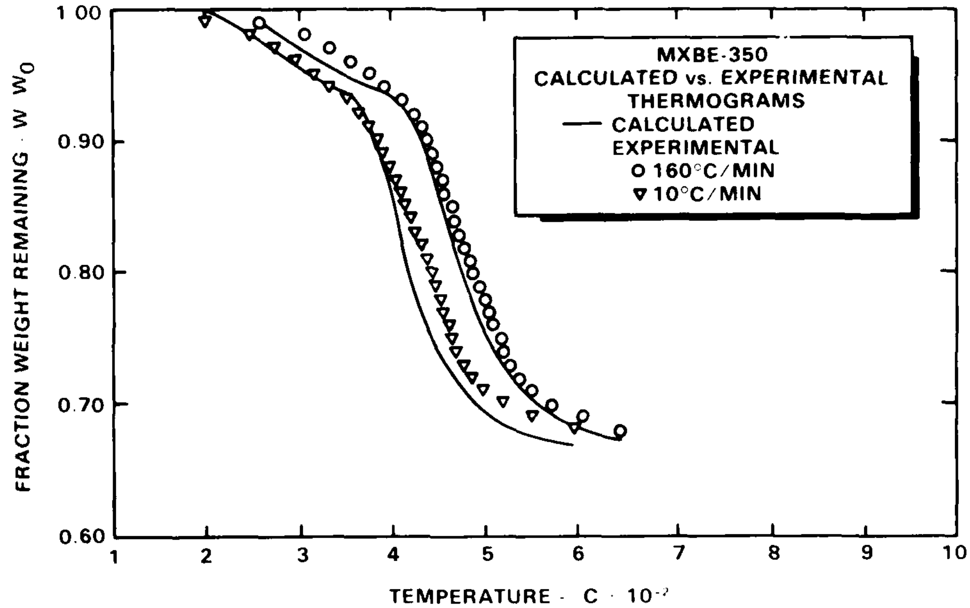

Figure 7.a Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for MXBE-350

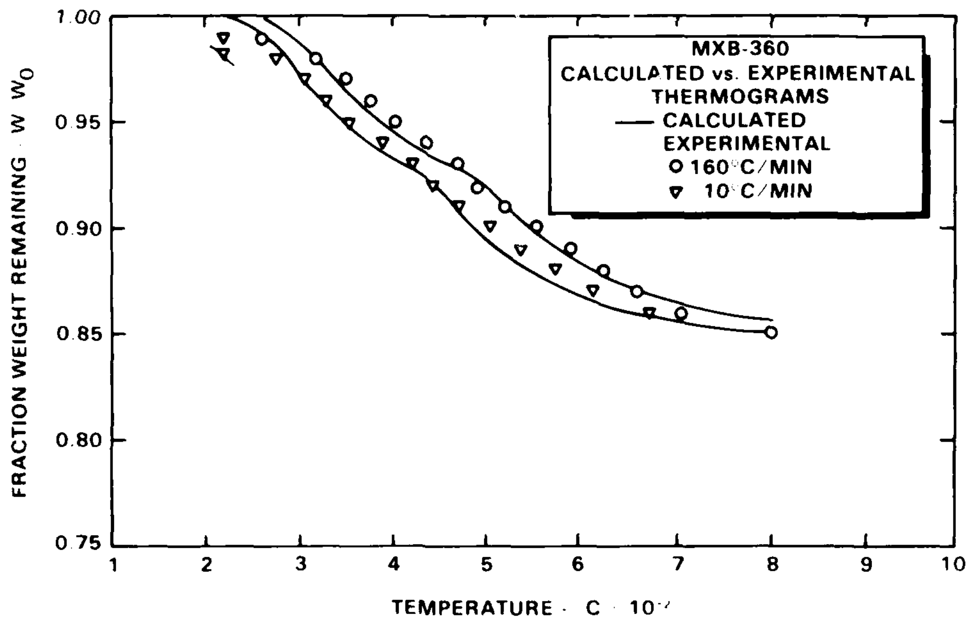

Figure 7.b Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for MXB-360

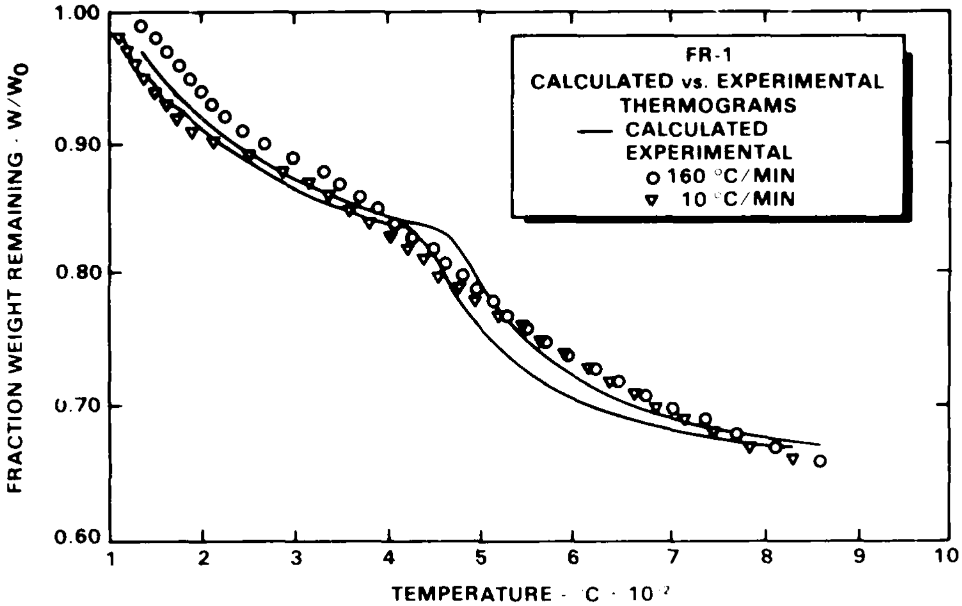

Figure 7.c Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for FR-1

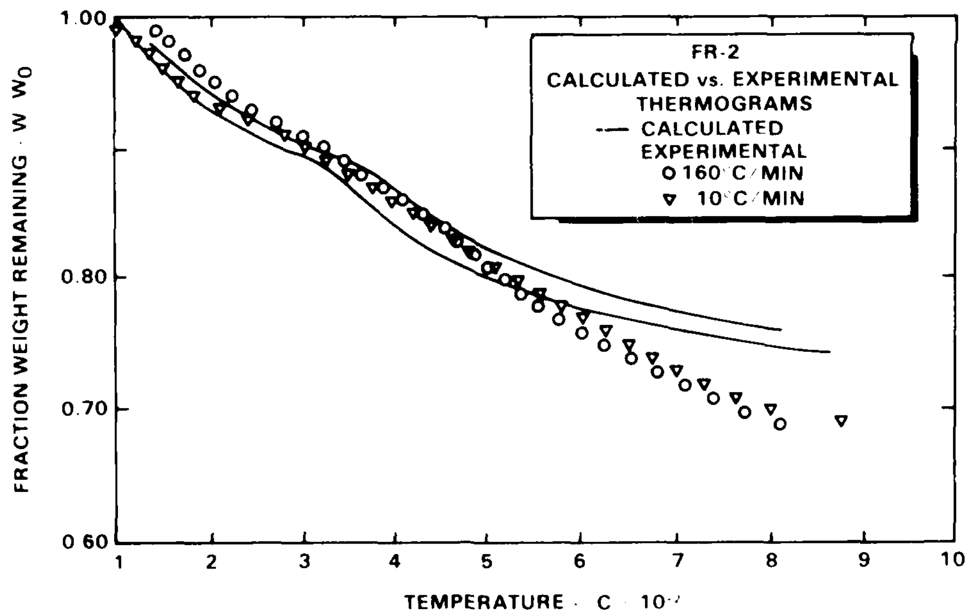

Figure 7.d Comparison of Calculated and Experimental Weight Loss Curves at 10 to 160°C/min for FR-2

DISCUSSION

As can be seen from the calculated versus experimental weight loss curves presented in Figure 7, the model predicts the weight loss with reasonable accuracy. The error in predicting the weight loss curves for MXBE-350, FR-1, and FR-2 could likely be reduced by separating the reaction into more than two regions, and determining a set of kinetic parameters for each region. Improving the fit of the lines to the data in Figure 5 leads to kinetic parameters that better predict the weight loss curve. The poor correlation of the calculated versus experimental weight loss curves at high temperatures for FR-2 in Figure 7d is the result of not utilizing experimentally deduced kinetic parameters in this region. Application of Friedman's method in this region led to negative activation energies, a result that is clearly not realistic in view of the physical meaning of activation energy. Therefore, the kinetic parameters determined from experimental data at lower temperatures and involving positive activation energies were used in the high-temperature region.

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APPENDIX

EXPERIMENTAL DATA

Weight Loss and Rate of Weight Loss Data for MXBE-350

w/w_0	Heating Rate 160°C/min w_0 = 7.8020		Heating Rate 100°C/min w_0 = 7.8451		Heating Rate 80°C/min w_0 = 7.9097		Heating Rate 40°C/min w_0 = 7.8589		Heating Rate 20°C/min w_0 = 7.9766		Heating Rate 10°C/min w_0 = 7.8000	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.99	255.9	0.0172	242.6	0.0121	234.0	0.0094	217.7	0.0046	212.2	0.0023	200.2	0.0010
0.98	305.5	0.0352	288.3	0.0245	294.0	0.0204	268.5	0.0104	257.5	0.0055	247.2	0.0029
0.97	334.4	0.0565	316.2	0.0388	314.0	0.0322	298.3	0.0168	284.5	0.0085	272.7	0.0042
0.96	[illegible]	0.0728	341.1	0.0482	335.0	0.0388	317.2	0.0197	304.8	0.0097	295.2	0.0049
0.95	376.1	0.0793	360.0	0.0507	355.0	0.0403	337.1	0.0201	326.0	0.0097	313.8	0.0050
0.94	391.9	0.0875	379.1	0.0553	374.1	0.0436	357.0	0.0219	345.3	0.0105	330.5	0.0053
0.93	407.7	0.1015	393.2	0.0704	388.1	0.0559	373.8	0.0277	362.3	0.0143	349.1	0.0068
0.92	419.2	0.1318	404.2	0.0905	401.2	0.0742	385.7	0.0382	373.7	0.0186	363.3	0.0092
0.91	428.7	0.1702	412.2	0.1120	408.2	0.0931	393.7	0.0463	385.2	0.0244	373.6	0.0114
0.90	432.9	0.1965	417.3	0.1370	415.3	0.1085	399.6	0.0550	390.4	0.0278	379.7	0.0132
0.89	439.2	0.2152	425.3	0.1546	423.3	0.1236	406.6	0.0615	397.6	0.0298	388.0	0.0145
0.88	444.5	0.2587	431.3	0.1706	427.3	0.1379	413.5	0.0667	407.0	0.0325	395.2	0.0156

Weight Loss and Rate of Weight Loss Data for MXBE-350 (Continued)

w/w_0	Heating Rate 160°C/min w_0 = 7.8020		Heating Rate 100°C/min w_0 = 7.8451		Heating Rate 80°C/min w_0 = 7.9097		Heating Rate 40°C/min w_0 = 7.8589		Heating Rate 20°C/min w_0 = 7.9766		Heating Rate 10°C/min w_0 = 7.8000	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.87	450.8	0.2775	435.3	0.1807	434.3	0.1440	418.5	0.0714	411.1	0.0342	401.4	0.0165
0.86	454.0	0.2947	440.4	0.1917	438.3	0.1508	424.4	0.0751	418.4	0.0365	408.6	0.0176
0.85	461.3	0.3111	444.4	0.1980	444.4	0.1560	429.4	0.0772	425.7	0.0390	412.7	0.0187
0.84	464.5	0.3233	451.4	0.2038	448.4	0.1602	435.3	0.0803	429.8	0.0409	418.9	0.0197
0.83	470.8	0.3298	455.4	0.2080	454.4	0.1644	440.3	0.0827	436.1	0.0433	423.0	0.0209
0.82	476.1	0.3397	460.4	0.2126	458.4	0.1679	444.3	0.0853	439.2	0.0450	430.2	0.0224
0.81	481.3	0.3454	465.5	0.2175	464.5	0.1702	447.2	0.0881	444.4	0.0464	433.3	0.0238
0.80	484.5	0.3495	471.5	0.2208	470.5	0.1741	453.2	0.0905	449.6	0.0469	439.5	0.0249
0.79	490.8	0.3527	474.5	0.2227	474.5	0.1757	457.2	0.0925	452.7	0.0464	442.6	0.0251
0.78	495.0	0.3561	478.5	0.2240	478.5	0.1757	463.1	0.0933	458.9	0.0449	448.7	0.0251
0.77	499.2	0.3561	484.5	0.2231	484.5	0.1735	466.1	0.0930	463.1	0.0419	450.8	0.0250
0.76	504.5	0.3552	487.6	0.2191	488.6	0.1663	472.0	0.0905	469.3	0.0374	457.0	0.0241

Weight Loss and Rate of Weight Loss Data for MXBE-350 (Continued)

w/w_0	Heating Rate 160°C/min w_0 = 7.8020		Heating Rate 100°C/min w_0 = 7.8451		Heating Rate 80°C/min w_0 = 7.9097		Heating Rate 40°C/min w_0 = 7.8589		Heating Rate 20°C/min w_0 = 7.9766		Heating Rate 10°C/min w_0 = 7.8000	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.75	512.9	0.3486	494.6	0.2116	495.6	0.1546	476.0	0.0853	475.5	0.2098	460.1	0.0226
0.74	516.0	0.3356	501.6	0.1967	502.6	0.1363	482.9	0.0763	484.9	0.0198	464.2	0.0200
0.73	523.4	0.3126	506.6	0.1628	510.6	0.1033	490.9	0.0616	498.4	0.0115	472.4	0.0158

Weight Loss and Rate of Weight Loss Data for MXB-360

w/w_0	Heating Rate 160°C/min, w_0 = 7.7894		Heating Rate 100°C/min, w_0 = 7.3376		Heating Rate 80°C/min, w_0 = 7.1716		Heating Rate 40°C/min, w_0 = 7.2935		Heating Rate 20°C/min, w_0 = 7.5161		Heating Rate 10°C/min, w_0 = 7.5213	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.98	320.0	0.0333	317.2	0.0245	314.2	0.0224	297.6	0.0111	298.6	0.0067	277.2	0.0027
0.97	351.0	0.0560	347.0	0.0379	343.1	0.0329	325.3	0.0162	326.3	0.0086	306.0	0.0040
0.96	377.3	0.0659	370.9	0.0407	368.2	0.0351	349.1	0.0173	348.1	0.0082	329.0	0.0041
0.95	402.6	0.0610	404.6	0.0308	394.5	0.0277	376.3	0.0150	382.4	0.0055	355.0	0.0029
0.94	439.1	0.0497	438.3	0.0313	429.9	0.0255	410.7	0.0125	418.5	0.0072	391.3	0.0029
0.93	469.4	0.0605	466.1	0.0397	454.2	0.0332	439.1	0.0167	442.0	0.0084	422.6	0.0040
0.92	493.7	0.0668	491.9	0.0413	482.6	0.0361	466.4	0.0175	469.5	0.0082	445.8	0.0041
0.91	521.1	0.0629	518.6	0.0357	505.9	0.0323	489.7	0.0151	497.1	0.0068	473.0	0.0035
0.90	553.5	0.0561	553.4	0.0319	535.2	0.0282	519.0	0.0134	527.7	0.0064	506.3	0.0031
0.89	589.9	0.0523	587.1	0.0313	568.7	0.0271	555.5	0.0130	562.5	0.0063	537.5	0.0031
0.88	624.2	0.0503	622.1	0.0304	598.9	0.0259	588.9	0.0122	601.3	0.0056	575.8	0.0029
0.87	660.3	0.0463	664.4	0.0249	635.3	0.0231	628.0	0.0106	647.2	0.0041	615.7	0.0024

Weight Loss and Rate of Weight Loss Data for FR-1

w/w_0	Heating Rate 160°C/min w_0 = 7.6956		Heating Rate 100°C/min w_0 = 7.8715		Heating Rate 80°C/min w_0 = 7.6218		Heating Rate 40°C/min w_0 = 7.7786		Heating Rate 20°C/min w_0 = 7.4357		Heating Rate 10°C/min w_0 = 7.5964	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.99	133.1	0.0472	125.5	0.0338	118.8	0.0297	108.0	0.0164	100.6	0.0106	93.7	0.0051
0.98	150.5	0.0830	142.3	0.0593	136.5	0.0546	125.7	0.0286	117.7	0.0169	110.0	0.0083
0.97	162.0	0.1089	156.1	0.0760	149.3	0.0678	137.5	0.0378	128.8	0.0207	120.2	0.0107
0.96	174.2	0.1292	166.9	0.0897	161.0	0.0773	146.3	0.0411	138.9	0.0221	129.4	0.0113
0.95	186.4	0.1403	177.8	0.0941	169.9	0.0794	156.1	0.0424	148.9	0.0218	138.6	0.0113
0.94	198.6	0.1420	198.6	0.0930	182.7	0.0773	166.0	0.0410	160.0	0.0199	150.8	0.0103
0.93	211.8	0.1361	204.4	0.0859	195.5	0.0706	177.8	0.0371	170.9	0.0175	162.0	0.0090
0.92	225.0	0.1245	217.1	0.0753	208.3	0.0613	191.5	0.0315	183.8	0.0147	173.7	0.0076
0.91	244.3	0.1055	232.9	0.0633	224.0	0.0513	207.3	0.0256	199.6	0.0114	190.1	0.0058
0.90	266.6	0.0807	255.5	0.0457	244.7	0.0375	227.0	0.0191	222.4	0.0081	212.4	0.0038
0.89	298.1	0.0561	286.1	0.0306	273.3	0.0248	254.6	0.0119	258.0	0.0046	252.2	0.0021
0.88	329.6	0.0617	317.6	0.0351	307.7	0.0255	294.9	0.0113	295.6	0.0066	288.1	0.0032

Weight Loss and Rate of Weight Loss Data for FR-1 (Continued)

w/w_0	Heating Rate 160°C/min w_0 = 7.6956		Heating Rate 100°C/min w_0 = 7.8715		Heating Rate 80°C/min w_0 = 7.6218		Heating Rate 40°C/min w_0 = 7.7786		Heating Rate 20°C/min w_0 = 7.4357		Heating Rate 10°C/min w_0 = 7.5964	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.87	346.9	0.0750	342.2	0.0470	333.3	0.0358	320.5	0.0171	319.4	0.0087	313.3	0.0042
0.86	368.1	0.0816	361.2	0.0490	353.0	0.0386	344.1	0.0184	341.1	0.0089	335.6	0.0042
0.85	387.3	0.0816	379.9	0.0485	375.4	0.0380	365.4	0.0181	365.1	0.0090	357.1	0.0043
0.84	407.4	0.0816	404.7	0.0492	394.8	0.0386	386.1	0.0188	387.4	0.0094	378.7	0.0047
0.83	425.6	0.0839	424.3	0.0516	416.3	0.0409	407.8	0.0200	408.7	0.0104	401.4	0.0051
0.82	445.8	0.0886	440.9	0.0556	436.7	0.0440	426.4	0.0217	426.9	0.0114	419.9	0.0055
0.81	460.9	0.0959	460.5	0.0601	454.1	0.0470	445.0	0.0238	441.1	0.0121	438.4	0.0057
0.80	479.0	0.1019	477.1	0.0638	471.4	0.0509	460.5	0.0248	459.3	0.0124	454.9	0.0057
0.79	493.2	0.1055	493.6	0.0652	487.8	0.0520	479.2	0.0253	476.5	0.0118	475.5	0.0053
0.78	511.3	0.1059	510.2	0.0639	504.1	0.0512	496.7	0.0244	491.7	0.0113	494.1	0.0049
0.77	527.4	0.1016	529.8	0.0593	520.4	0.0479	513.3	0.0226	514.0	0.0101	518.8	0.0045
0.76	547.6	0.0929	548.4	0.0544	541.9	0.0433	536.0	0.0203	535.2	0.0093	542.5	0.0043

Weight Loss and Rate of Weight Loss Data for FR-1 (Continued)

w/w_0	Heating Rate 160°C/min w_0 = 7.6956		Heating Rate 100°C/min w_0 = 7.8715		Heating Rate 80°C/min w_0 = [illegible]		Heating Rate 40°C/min w_0 = [illegible]		Heating Rate 20°C/min w_0 = 7.44[illegible]		Heating Rate 10°C/min w_0 = 7.5464	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.75	567.8	0.0837	571.2	0.0488	563.5	[illegible]	[illegible]	[illegible]	[illegible]	0.0089	564.1	0.0043
0.74	592.0	0.0754	596.0	0.0447	588.[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	0.0087	[illegible]	0.0043
0.73	620.0	0.0694	621.7	0.0427	612.6	[illegible]	[illegible]	[illegible]	[illegible]	0.0087	614.0	0.0043
0.72	645.0	0.0664	647.4	0.0415	639.[illegible]	[illegible]	[illegible]	[illegible]	634.8	0.0084	637.0	0.0042
0.71	673.0	0.0647	676.1	0.0412	665.5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	662.0	0.0039
0.70	702.0	0.0641	703.8	0.0409	690.0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	686.0	0.0036
0.69	737.0	0.0615	740.4	0.0381	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	715.0	0.0033
0.68	770.0	0.0564	770.1	0.0334	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	747.0	0.0030
0.67	811.1	0.0495	813.9	0.0294	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	783.4	0.0025

Weight Loss and Rate of Weight Loss Data for FR-2

w/w_0	Heating Rate 160°C/min w_0 = 7.3750		Heating Rate 100°C/min w_0 = 7.3284		Heating Rate 80°C/min w_0 = 7.5891		Heating Rate 40°C/min w_0 = 7.7749		Heating Rate 20°C/min w_0 = 7.6085		Heating Rate 10°C/min w_0 = 7.7427	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.99	139.9	0.0450	130.4	0.0322	128.6	0.0290	115.9	0.0139	109.7	0.0085	101.9	0.0043
0.98	156.2	0.0748	151.1	0.0554	147.3	0.0472	134.5	0.0242	127.8	0.0138	121.2	0.0068
0.97	173.2	0.0974	166.9	0.0680	164.0	0.0571	148.3	0.0293	139.9	0.0154	134.5	0.0077
0.96	187.4	0.1087	180.7	0.0730	178.8	0.0577	164.0	0.0300	153.9	0.0150	149.8	0.0074
0.95	203.6	0.1094	196.5	0.0708	193.5	0.0535	177.8	0.0270	169.9	0.0133	164.0	0.0063
0.94	222.9	0.1014	210.3	0.0643	209.3	0.0453	196.5	0.0221	186.8	0.0110	183.4	0.0049
0.93	243.3	0.0866	230.9	0.0542	230.9	0.0364	219.1	0.0167	209.5	0.0084	209.5	0.0035
0.92	270.7	0.0675	255.5	0.0423	260.5	0.0262	253.6	0.0113	242.2	0.0057	242.5	0.0023
0.91	300.2	0.0588	285.1	0.0356	296.9	0.0269	284.1	0.0117	279.8	0.0062	279.3	0.0032
0.90	323.5	0.0679	307.7	0.0413	320.5	0.0353	312.6	0.0163	306.5	0.0083	302.6	0.0040
0.89	344.9	0.0793	328.4	0.0498	342.2	0.0377	335.3	0.0176	330.2	0.0085	326.9	0.0039
0.88	363.1	0.0796	349.1	0.0504	363.2	0.0345	358.2	0.0161	354.0	0.0080	349.2	0.0039

Weight Loss and Rate of Weight Loss Data for FR-2 (Continued)

w/w_0	Heating Rate 160°C/min w_0 = 7.5750		Heating Rate 100°C/min w_0 = 7.3284		Heating Rate 80°C/min w_0 = 7.5891		Heating Rate 40°C/min w_0 = 7.7749		Heating Rate 20°C/min w_0 = 7.6085		Heating Rate 10°C/min w_0 = 7.7427	
	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)	T(°C)	$(-1/w_0)(dw/dt)$ (1/min)
0.87	386.3	0.0731	372.6	0.0461	385.7	0.0339	384.0	0.0165	378.3	0.0084	375.6	0.0041
0.86	406.4	0.0738	394.5	0.0458	411.2	0.0364	405.7	0.0177	399.6	0.0091	398.3	0.0045
0.85	428.6	0.0776	415.0	0.0486	432.6	0.0401	429.5	0.0199	419.8	0.0100	420.9	0.0050
0.84	448.8	0.0835	435.7	0.0524	449.0	0.0441	448.1	0.0219	439.1	0.0110	439.5	0.0052
0.83	465.9	0.0904	455.4	0.0574	467.3	0.0476	466.7	0.0233	459.3	0.0113	461.1	0.0052

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